



# **Development Of Partially Fluorinated Resin Apex Seals**

**Contract NAS3-23054**

**H. E. Green, G. E. C. Chang, S. H. Powell and K. Yates**

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Prepared for  
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1. Report No. NASA CR-174706		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle DEVELOPMENT OF PARTIALLY FLUORINATED RESIN APEX SEALS				5. Report Date March 1, 1984	
				6. Performing Organization Code	
7. Author(s) H. E. Green, G. E. C. Chang, S. H. Powell, and K. Yates				8. Performing Organization Report No. 38603-6009-UT-00	
				10. Work Unit No.	
9. Performing Organization Name and Address TRW Energy Development Group Redondo Beach, CA 90278				11. Contract or Grant No. NAS3-23054	
				13. Type of Report and Period Covered	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D. C. 20546				14. Sponsoring Agency Code	
15. Supplementary Notes					
16. Abstract <p>Samples of TRW's partially fluorinated polyimides were prepared and molded in the form of discs and pins for test as potential apex seal materials for advanced rotary combustion engines. The polyimides were formulated from the diamine 2,2-bis 4-(4-aminophenoxy)phenyl hexafluoropropane (4-BDAF) and the dianhydrides of pyromellitic acid (PMDA) and benzophenonetetracarboxylic acid (BTDA). Test specimens were made with 4-BDAF/PMDA, 4-BDAF/80 PMDA, 20 BTDA and 4-BDAF/60 PMDA, 40 BTDA, with and without carbon fiber reinforcement; a total of twelve formulations.</p> <p>Tribological testing was performed at Lewis Research Center at sliding speeds of 0.31 to 11.6 m/s and at temperatures of from 298K to 573K. The results showed that the carbon fiber filled polyimides, particularly the 80/20 compositions, have an excellent balance of wear/friction at 573K. The results of the tests with the unfilled, 80/20 and 60/40 compositions indicate an unusual combination of high friction and low wear which may be advantageous in such applications as brakes and traction drives.</p>					
17. Key Words (Suggested by Author(s)) Rotary Engine, Apex Seals, Polyimides			18. Distribution Statement Unclassified - Unlimited		
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of pages	
				22. Price*	

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## FOREWORD

This report was prepared by TRW Electronics and Defense Sector under Contract NAS3-23054, "Development of Partially Fluorinated Resin Apex Seals." The technical effort was conducted during 1982 and 1983 under sponsorship of NASA Lewis Research Center. Mr. William F. Hady served as the NASA Program Monitor. Dr. Robert L. Fusaro generated and interpreted the tribological data. The project team at TRW comprised Dr. Howard E. Green, program manager, Dr. Glenn E.C. Chang, principal investigator, Mr. Kenneth Yates, monomer and polymer preparation and Mr. Stephen H. Powell, who was responsible for resin fabrication studies. Monomer and Polymer characterization work was conducted under the direction of Mr. Michael K. O'Rell.

## SUMMARY

This final report document describes work performed by the TRW Electronics and Defense Sector for the National Aeronautics and Space Administration, Lewis Research Center, under contract NAS3-23054. The technical work was performed during the period of September, 1982 through June, 1983.

The objective of the program was to prepare samples of TRW's partially fluorinated polyimides in several versions for test by NASA as candidate materials for apex seals an advanced rotary combustion engine.

During this project molding powders of three versions of the partially fluorinated polyimides, with and without carbon fiber reinforcement were prepared and molded in the form of discs and machined bullet riders.

Tribological testing at Lewis Research Center showed that some versions the TRW materials exhibited unusually low wear rates at 298K and at 573K as well as very good frictional coefficients.

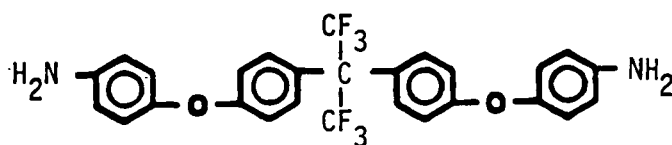


## 1.0 INTRODUCTION

This final report documents the work performed by TRW Electronics and Defense Sector during 1982 and 1983 on the Development of Partially Fluorinated Resin Apex Seals. This work was sponsored by the National Aeronautics and Space Administration, Lewis Research Center, under contract NAS3-23054.

The objective of this program was to provide NASA with samples of TRW's partially fluorinated polyimides (PFPI) in appropriate configurations to test and evaluate as potential apex seal materials for advanced rotary combustion engines.

The PFPIs are based on combinations of aromatic dianhydrides with TRW's partially fluorinated aromatic diamine 2,2-bis[4-(4-aminophenoxy)-phenyl]hexafluoropropane (4-BDAF, see structure below). Polyimides incorporating 4-BDAF had shown great promise in previous work at NASA/LeRC for use as rotary engine apex seals.



4-BDAF

During this program 12 filled and unfilled candidate seal materials were prepared using three types of carbon fiber and three versions of the partially fluorinated polyimides. Molding compounds were prepared from the candidate seal materials and then compression molded into forms that were machined into tribological test specimens. A total of 36 specimens were delivered to NASA for testing and evaluation. Table I shows the types and numbers of materials that were prepared for study.

The tribological tests performed at NASA/LeRC revealed that the TRW materials when filled with carbon fibers had frictional coefficients similar to that of other polyimides at 298K and 573K but much lower wear rates. At 298K the unfilled 80/20 and 60/40 versions of the PFPIs had a surprising combination of high friction and low wear properties.

TABLE I  
TEST SPECIMENS

Dianhydride <sup>a</sup>	Type of Carbon Fiber <sup>b</sup>			
	None	Celanese GY-70	Union Carbide Pitch Fiber VSB-160	Union Carbide Thornel 50
100% PMDA	3 riders 1 disc	3 riders 1 disc	3 riders 1 disc	3 riders 1 disc
80% PMDA & 20% BTDA	3 riders 1 disc	3 riders 1 disc	3 riders 1 disc	3 riders 1 disc
60% PMDA & 40% BTDA	3 riders 1 disc	3 riders 1 disc	3 riders 1 disc	3 riders 1 disc

<sup>a</sup>PMDA = pyromellitic acid dianhydride  
BTDA = benzophenonetetracarboxylic acid dianhydride.

<sup>b</sup>All composites are 50% by weight resin solids and 50% by weight carbon fiber solids.

The remainder of this final report is divided into the following sections.

- 2.0 Preparation of Polymers
- 3.0 Sizing and Molding Powder Preparation
- 4.0 Molding Studies and Specimen Preparation
- 5.0 Results of Tribological Studies
- 6.0 Conclusions and Recommendations

## 2.0 PREPARATION OF POLYMERS

The initial technical effort of the project was concentrated on determining the best polymerization method of preparing the mixed polyimide resins for molding powders. Inherent viscosity measurements and gel permeation chromatography were the analytical tools employed in the determination of the method which afforded the greatest molecular weight and narrowest molecular weight distribution; two parameters associated with the optimization of polymer performance. The two methods tested involved: 1) mixing appropriate proportions of polyamide acid solutions of 100% 4-BDAF/PMDA and 100% 4-BDAF/BTDA ("mixed varnish" method) and 2) preparing a separate solution for each mixture by adding the respective anhydrides, in the correct proportions, to the 4-BDAF during the polymerization ("mixed anhydride" method).

The test reactions were performed on a small (0.01 mole) scale to minimize the hydrolysis that might occur during the addition of larger quantities of reagents. The polymerizations performed in 25% by weight solids solutions proved to be difficult to handle because of very high viscosity, so the standard concentration for the test reactions was fixed at 15% by weight solids.

In general, the polymerization procedure involved the addition of the anhydride or a mixture of anhydrides to a solution of 4-BDAF in dimethylsulfoxide (DMSO) while maintaining the reaction mixture at less than 298K (77°F). The mixture was stirred for one additional hour after it appeared to be homogeneous. Samples of the resultant varnishes were submitted for analysis immediately, while the remaining portions were placed in a vacuum oven to remove solvent to prevent hydrolysis.

The inherent viscosity measurement of the test reactions are shown in Table II. The results indicate that the "mixed anhydrides" method affords polyamide acids with greater molecular weight, although the viscosities obtained by both methods are indicative of high molecular weight (estimated to be  $>30,000$  g/mol on the basis of literature values).

The gel permeation chromatography (gpc) was performed with a Waters 150C Automatic Liquid Chromatograph/Gel Permeation Chromatograph equipped with Waters  $10^3\text{\AA}$ ,  $10^4\text{\AA}$  and  $10^5\text{\AA}$  Microstyrogel columns which had been packed in dimethylformamide (DMF). The eluting solvent was DMF (at 1.0 ml/min). The sample concentration was 0.1% and the column temperature 308K (95°F).

The results of the gpc analyses confirmed the viscosity results (i.e., that the mixed anhydride method produced resins with greater molecular weight) when the samples were analyzed within one hour. There were no significant differences in molecular weight distribution among the samples. However, when the samples were analyzed after longer time periods the chromatograms showed an apparent increase in the molecular weight and narrowing of the molecular weight distribution. An example of this phenomenon is shown in Figures 1 and 2. The chromatograph shown in Figure 1, run within one hour of sample preparation, displays a peak maximum at 1059 seconds. The peak maximum in Figure 2, run after 24 hrs is at 999 seconds. Since gpc is a "size exclusion" analysis, shorter elution time usually indicates greater molecular weight.

A possible explanation of these observations may lie in the polar nature of the polyamide-acid molecule. It is expected that each polyamide-acid chain is associated with a "cloud" of polar solvent molecules. As hydrolysis proceeds, the number of free acid and amine chain ends increases, further enhancing the polar nature of the chains and, consequently, the size of the closely held "cloud" that surrounds them. In this way, a lower molecular weight, but more polar molecule will pass through a non-polar Styrogel column more rapidly than a higher molecular weight molecule since its apparent size, chain plus solvent cloud, is greater. The apparent

molecular weight distribution would be expected to narrow as hydrolysis proceeds since the limit will approach monomer chain length with time.

In sum it appears that gpc is not a reliable analytical tool for polar polymers such as polyamide-acids in polar solvents such as DMF.

TABLE II  
POLYMERIZATION METHOD TESTS

% Anhydride <sup>a</sup>		Method <sup>b</sup>	Inherent Viscosity <sup>c</sup> (dL/g)
PMDA	BTDA		
100	0	--	1.5801
0	100	--	1.3329
80	20	M.A.	1.5234
80	20	M.V.	1.4688
60	40	M.A.	2.0640
60	40	M.V.	1.4301

<sup>a</sup>Diamine = 4-BDAF

<sup>b</sup>M.A. = mixed anhydride  
M.V. = mixed varnish

<sup>c</sup>303K, 0.5 g/dL in DMSO

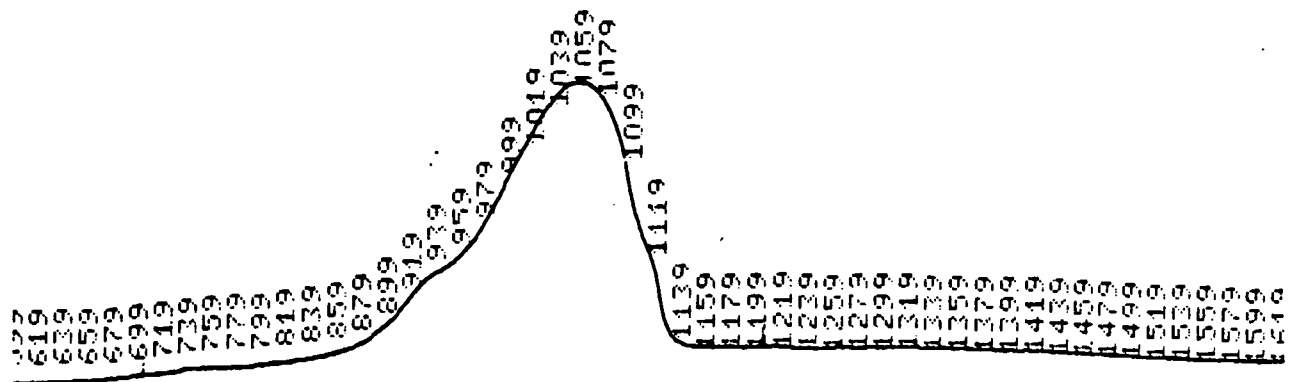


Figure 1. GPC of (60/40) (PMDA/BTDA) - 4-BDAF, 1 hr.

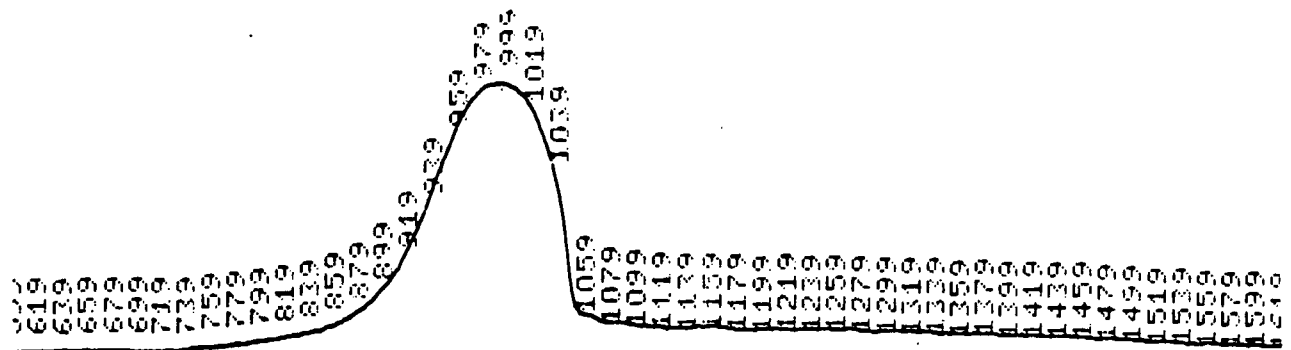


Figure 2. GPC of (60/40) (PMDA/BTDA) - 4-BDAF, 24 hr.

The selected "mixed anhydride" method was used to prepare larger, 250g-size batches of the three base resins. Briefly, the polymerization involved the addition of the anhydride or a mixture of anhydrides to a solution of an equimolar quantity of 4-BDAF in dimethylsulfoxide (DMSO) while maintaining the reaction mixture at less than 298K (77°F). The solution was stirred for one additional hour after it appeared to be homogeneous. These larger scale reactions provided polyamide-acids with the following inherent viscosities [0.5% solutions in DMSO at 303K (86°F)]:

4-BDAF/PMDA = 1.27 dL/g

4-BDAF/80 PMDA:20 BTDA = 0.97 dL/g

4-BDAF/60 PMDA:40 BTDA = 0.91 dL/g

It is not surprising that these viscosities are lower than those obtained in the small scale polymerization method tests shown in Table II. Handling larger quantities of moisture sensitive reagents provides greater opportunity for molecular weight-reducing hydrolysis. However, the inherent viscosities obtained for the larger runs are indicative of high molecular weight polymers appropriate for use in compression molding.

The polyamide-acid solutions similar to those described above were converted to dry powders by removing the solvent under vacuum at 358 to 373K (185 to 212°F) for at least 12 hours. They were then converted to polyimides by heating under vacuum for an additional 15 hours at 477K (400°F). The imidization time and temperature were based on isothermal thermal gravimetric analysis (tga) data which showed that imidization is essentially complete at 477K (400°F) after six hours. Infra red spectra (ir) indicated that 4-BDAF-based polyimides were completely imidized (the spectra of imidized 4-BDAF/PMDA, 4-BDAF/80 PMDA:20 BTDA and 4-BDAF/60 PMDA:40 BTDA are shown in Figures 3, 4 and 5, respectively) under these conditions; however compression molding studies performed subsequently showed that longer heating times are required to remove all of the DMSO polymerization solvent.

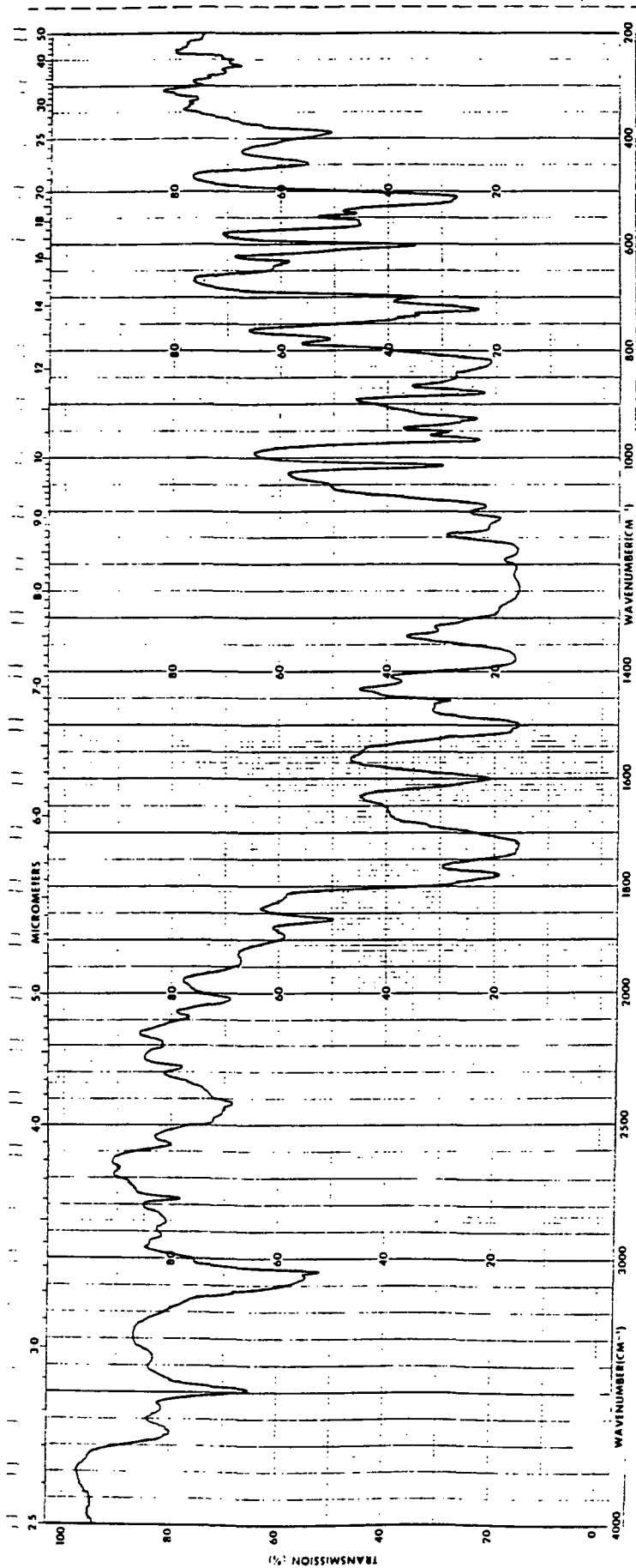


Figure 3. 4-BDAF/PMDA



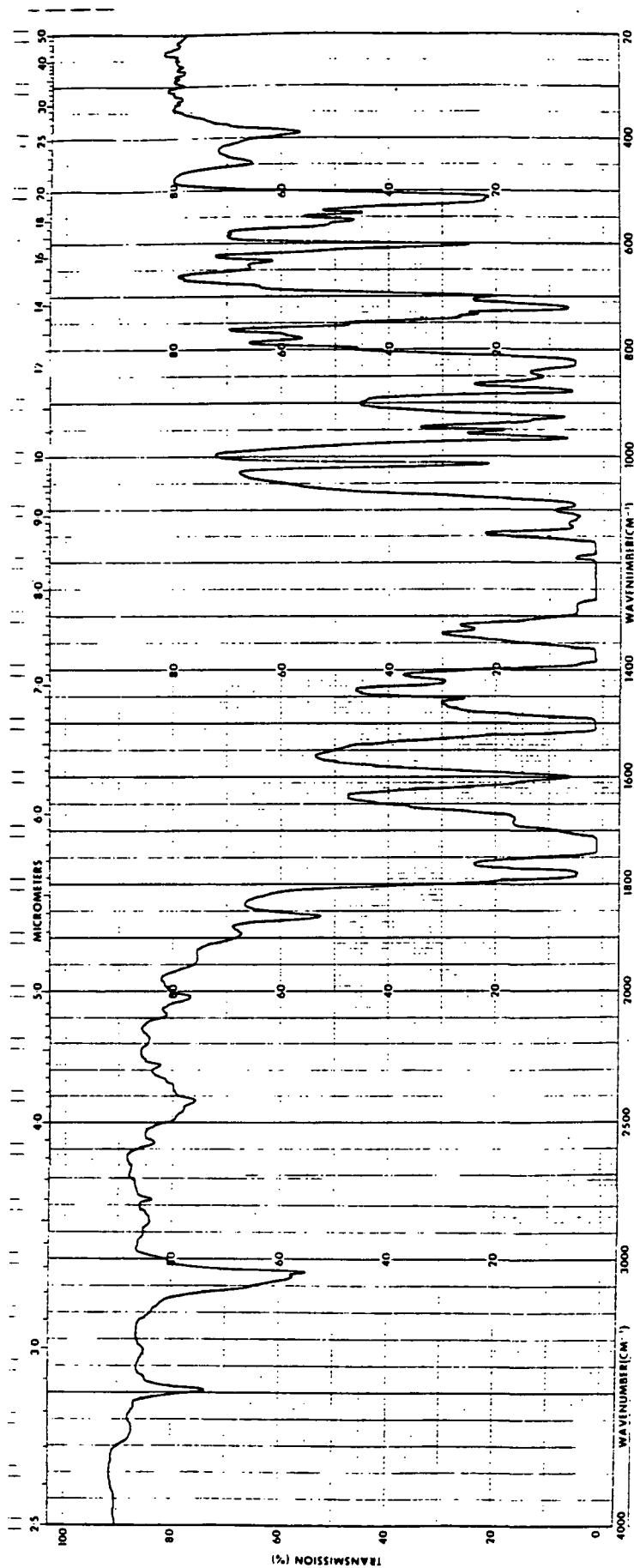


Figure 4. 4-BDAF/80 PMDA:20 BTDA

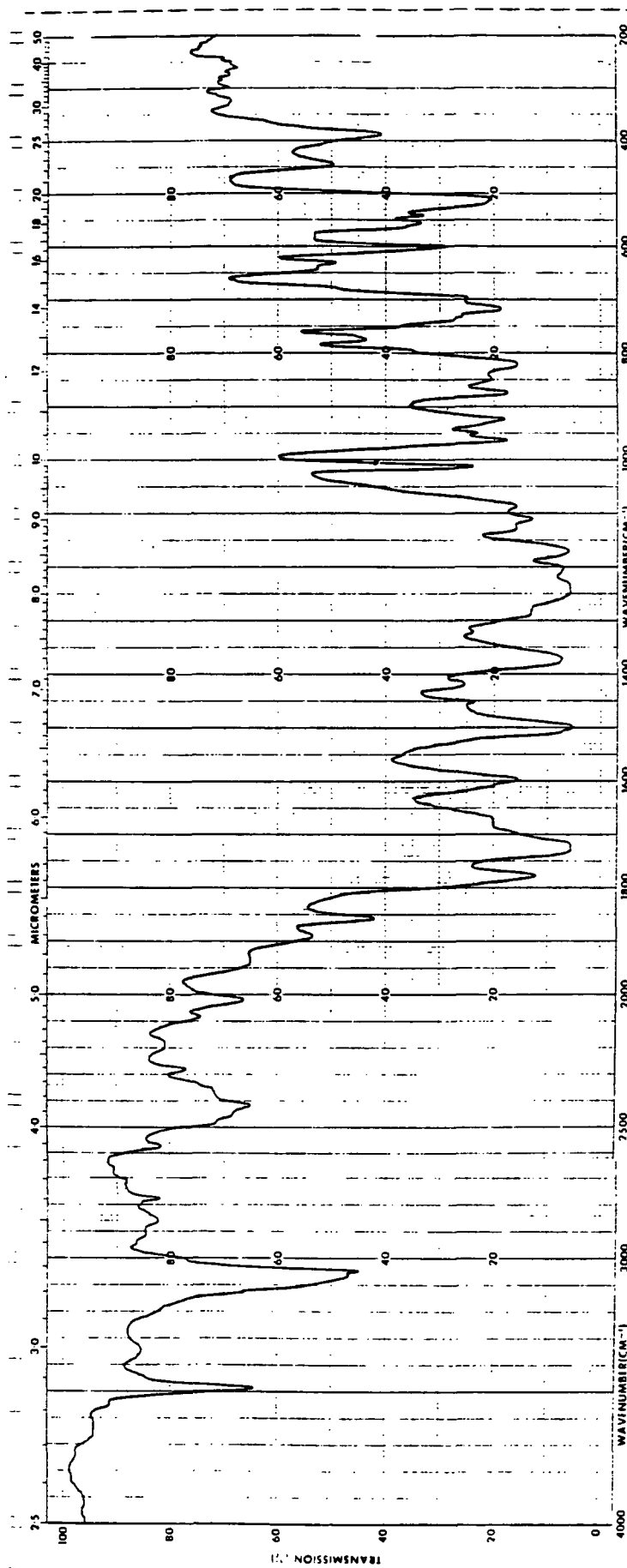


Figure 5. 4-BDAF/60 PMDA:40 BTDA

### 3.0 SIZING AND MOLDING POWDER PREPARATION

A fiber sizing procedure was established using Celion 6000 chopped fibers which had been delivered with a "polyimide compatible" size. The fibers were soaked in DMSO for two hours. The DMSO turned dark, indicating that the original size is at least partially soluble in the solvent. The fibers were recovered by filtration. The damp fibers were placed in a round bottomed flask and suspended in a DMSO solution of 4-BDAF/PMDA which contained sufficient solids to provide a 1% by weight coating of polymer on the fibers. The DMSO was removed under vacuum on a rotary evaporator at 373K (212°F). The dry fibers were then subjected to imidization at 477K (400°F) for approximately 12 hours. The resulting fibers were free flowing. This procedure was employed to size samples of the other graphite fibers.

Two molding powder preparation methods were investigated. The "wet mix" method comprised the addition of sized fibers to an amide-acid solution which was subsequently dried and imidized. The "dry mix" method involved the addition of the fibers to dry, imidized resin followed by prolonged mixing by a variety of mechanical methods.

Microscopic examination of pieces molded from powders prepared by both methods clearly showed that the "wet mix" method resulted in more uniform distribution of the fibers in the matrix and that method was used for the fabrication of deliverables.

The efficacy of the PFPI sizing and mixing methods was tested by molding test specimens (see Section 4.0 for molding procedures). Scanning Electron Microscope photomicrographs were obtained of broken sections of molded discs of 50:50 (by weight) 4-BDAF/PMDA:Union Carbide VSB fiber sized with either 4-BDAF/PMDA or the as received, commercial, "polyimide compatible" sizing. Several of the photomicrographs are reproduced in Figures 6 through 9. A comparison of Figures 6 and 7 (TRW size) and Figures 8 and 9 (commercial size) strongly supports the decision to size all of the fibers to be used in preparing the discs and bullet riders with the TRW material. The photomicrographs clearly show that the 4-BDAF/PMDA matrix resin wets the fibers more completely when they are sized with the TRW resin.



Figure 6. SEM of Molded Disc, 50:50 (by weight)  
4-BDAF/PMDA:Union Carbide VSB-160  
(sized with 4-BDAF/PMDA)  
Magnification 500X

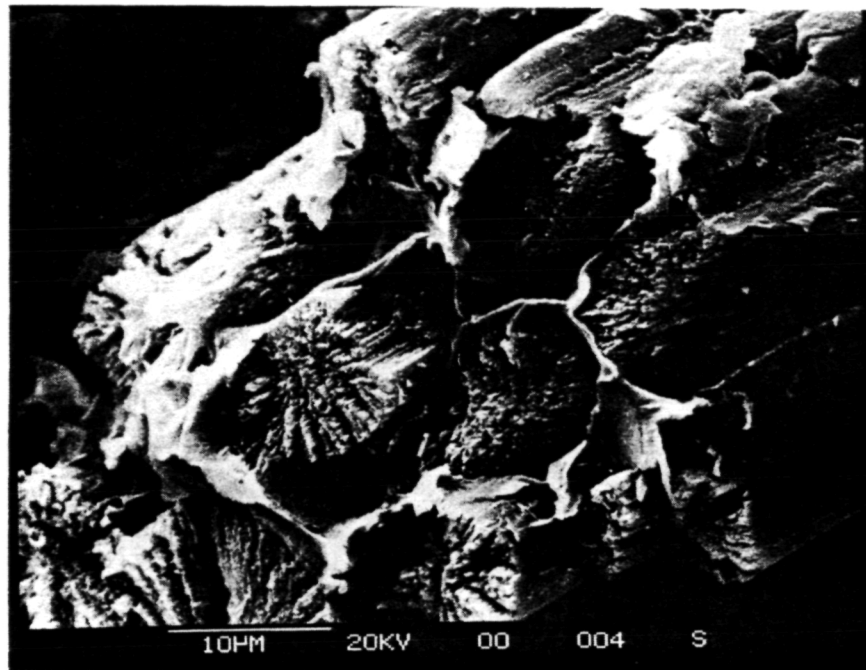


Figure 7. SEM of Molded Disc, 50:50 (by weight)  
4-BDAF/PMDA:Union Carbide VSB-160  
(sized with 4-BDAF/PMDA)  
Magnification 2000X

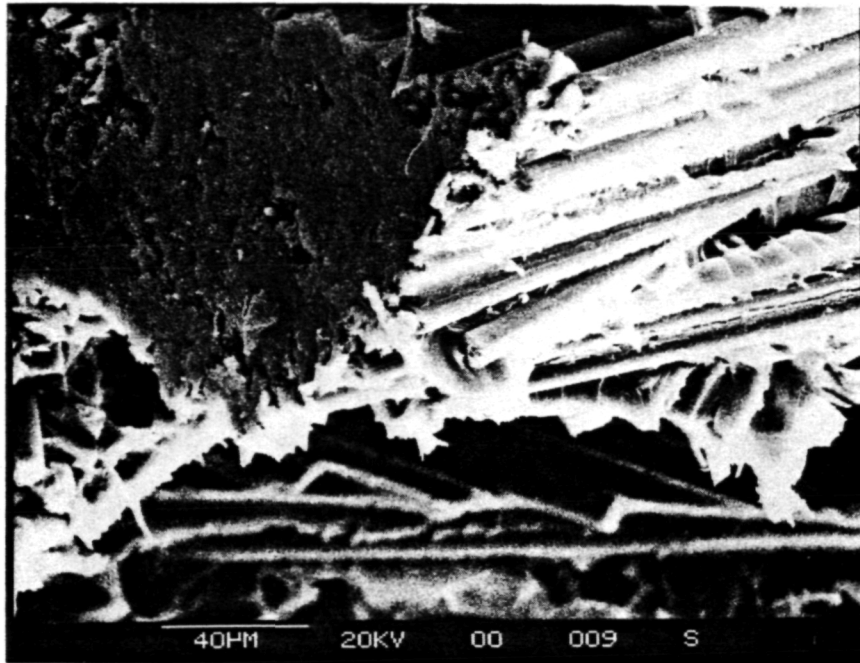


Figure 8. SEM of Molded Disc, 50:50 (by weight)  
4-BDAF/PMDA:Union Carbide VSB-160 fiber  
(commercial size)  
Magnification 500X

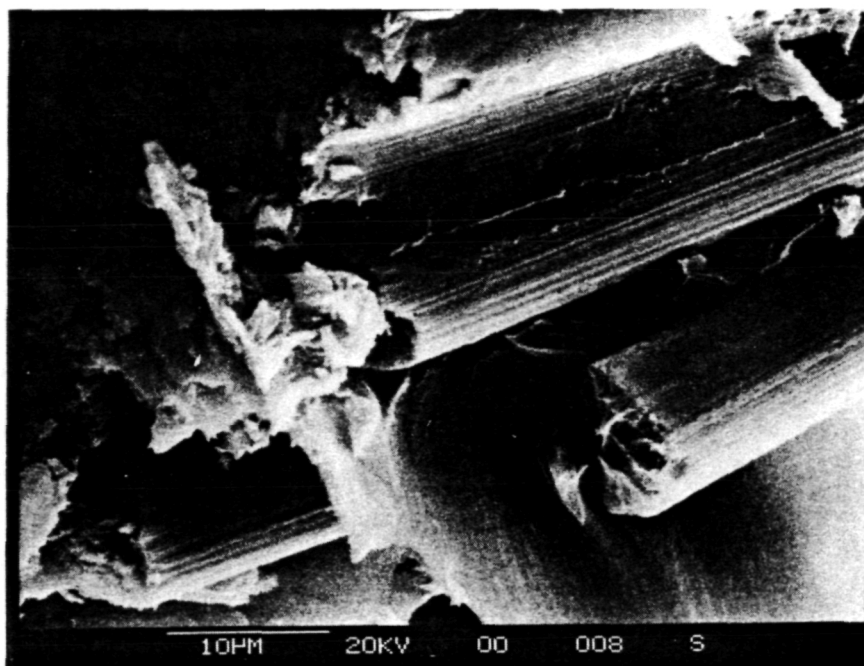


Figure 9. SEM of Molded Disc, 50:50 (by weight)  
4-BDAF/PMDA:Union Carbide VSB-160 fiber  
(commercial size)  
Magnification 2000X

#### 4.0 MOLDING STUDIES AND SPECIMEN PREPARATION

Molding studies were undertaken to select a temperature/pressure schedule to provide molded polyimide blanks from which test discs and pins could be machined.

Thermal gravimetric analysis (tga), performed in the isothermal mode indicated that six hours at 477K (400°F) was sufficient to result in complete imidization (*vide supra*). However, molding powders prepared by imidizing at 477K for ~ 15 hours formed voided compression molded discs and evolved a strong odor of the polymerization solvent, dimethyl sulfoxide (DMSO). Cure at 477K (400°F) followed by additional heating at 644K (700°F) for one hour resulted in incomplete consolidation of the molded parts, indicating excessive cure. On the basis of neat resin moldings the best cure temperature appeared to be four to six hours at 573K (572°F) (isothermal tga data showed that two hours at 573K was sufficient to remove all volatiles).

The standard molding procedure, established after examination of neat resin moldings fabricated under a variety of schedules, was as follows:

The charge was added to a hot mold [ $\sim$  760K (520°F)] and the mold was heated to 644K (700°F) over 20 min. The mold was then closed and 41.3 PMa (6000 psi) pressure was applied and periodically relieved ("bumped") as the temperature was increased over a 15 min period to 727K (850°F). The pressure and temperature were then held at 41.3 MPa and 727K for a 30 min period. The mold was then allowed to cool to ca. 630K (650°F) before the pressure was allowed to decay. The tool was opened when its temperature had reached 477K (400°F).

Molding powders were prepared using GY-70, VSB-160 and Thorne1 50 fibers which had been washed with DMSO and then sized with 4-BDAF/PMDA according to the methods previously described. The molding powders were used to mold 0.057m x 0.006m (2.25 in x 0.25 in) and 0.057m x 0.013m (2.25 in x 0.5 in) discs. After a 16 hr postcure at 630K (675°F), the thinner discs were machined to give parallel surfaces and the required hole was drilled in the center of each disc perpendicular to the surfaces. The thicker discs were machined into pins.



In several instances voids were discovered in the molded parts while they were being machined. The presence of voids was more prevalent in discs prepared with the mixed resins (i.e., 4-BDAF/60:40 PMDA:BTDA and 4-BDAF/80:20 PMDA:BTDA), but there was no obvious pattern with respect to fiber type. The voids were primarily in the interior of the center of the thicker discs. Since the bullet riders were machined such that the finished ends were toward the outer edge of the discs it was possible to obtain a number of riders of each type which have ends that will permit tribological testing. Extra bullet riders (6 to 10 of each instead of the required 3) were delivered to permit selection of the best specimens for test. The cause(s) of the voids is not clear at this time. However, several possible causes are suggested by the location of the voids and the fact that the neat-resin discs were not voided. In all likelihood, additional experimentation will be required to define the optimum molding conditions, both with respect to temperature and pressure, and fiber loading level for each of the resin systems in order to obtain void-free parts.

## 5.0 RESULTS OF THE TRIBOLOGICAL STUDIES

The machined test specimens were sent to NASA Lewis Research Center where they were tested by R. L. Fusaro using a pin-on-disc test apparatus. The tests were performed at 298K (77°F) and 573K (572°F) using a standard 1000 rpm rotational speed and a one kilogram load (see below for an exception to the standard rotational speed).

The composite pins were run against two different metal surfaces; Rene 41 and Haynes 6B. The metal alloys gave different wear rates (Table III) and coefficients of friction (Table IV), perhaps due to differences in extent of oxidation at 573K and/or surface texture. Unfortunately, the data do not show a pattern. All subsequent data is presented as an average of the values obtained for both counterfaces.

At the time of this writing all of the data for wear and friction at 298K were not available. The available data, displayed in Tables V and VI, respectively, show unusually high frictional coefficients ( $>0.95$ ) and low wear rates ( $7 \times 10^{-15} \text{ M}^3/\text{M}$ ) for the unfilled 80/20 and 60/40 compositions. A commercially available polyimide shows a much more usual combination of properties; frictional coefficient, 0.50 and wear rate,  $75 \times 10^{-15} \text{ M}^3/\text{M}$ . The filled PFPI compositions show very good combination of properties with low wear and low friction.

The wear (Table VII) and friction (Table VIII) data at 573K (averaged for counterface) show that the PFPIs have an excellent combination of properties relative to the few other materials capable of performing at 573K.

The effect of fiber type on wear and friction is shown in Figures 10 and 11, respectively. The data indicate that VSB-160, a pitch fiber, has some advantage in wear rate over the others. The reason of this difference is not immediately obvious.

Figure 12 shows the effect of rotational speed on the friction coefficient of 100% 4-BDAF/PMDA filled with GY-70 at 573K. There appears to be remarkably little change in frictional coefficient up to 4000 rpm.

TABLE III  
AVERAGE WEAR RATES ( $M^3/M \times 10^{-15}$ )  
VS. COUNTERFACE AT 573K

Fiber	Counterface	Resin Composition (%PMDA/%BTDA)		
		100	80/20	60/40
Thorne1 50	Rene 41	68	65	73
	Haynes 6B	62	75	65
GY-70	Rene 41	39	36	38
	Haynes 6B	47	34	36
VSB-160	Rene 41	37	28	36
	Haynes 6B	44	27	41

TABLE IV  
AVERAGE FRICTION COEFFICIENT VS. COUNTERFACE AT 573K

Fiber	Counterface	Resin Composition (%PMDA/%BTDA)		
		100	80/20	60/40
Thorne1 50	Rene 41	.13	.18	.26
	Haynes 6B	.34	.23	.18
GY-70	Rene 41	.22	.21	.14
	Haynes 6B	.36	.15	.14
VSB-160	Rene 41	.27	.20	.18
	Haynes 6B	.33	.18	.31

TABLE V

AVERAGE WEAR RATES ( $M^3/M \times 10^{-15}$ ) AT 298K

Fiber	Resin Composition (%PMDA/%BTDA)		
	100	80/20	60/40
Thornel 50	0.13	-- <sup>a</sup>	--
GY-70	0.40	--	--
VSB-160	0.10	--	0.093
None	300	7	7

---

<sup>a</sup>Data not Available

TABLE VI

AVERAGE FRICTION COEFFICIENT AT 298K

Fiber	Resin Composition (%PMDA/%BTDA)		
	100	80/20	60/40
Thornel 50	0.22	-- <sup>a</sup>	--
GY-70	0.26	--	--
VSB-160	0.25	--	0.26
None	0.50	0.95	0.95

---

<sup>a</sup>Data not Available

TABLE VII

AVERAGE WEAR RATES ( $M^3/M \times 10^{-15}$ ) AT 573K

Fiber	Resin Composition (%PMDA/%BTDA)		
	100	80/20	60/40
Thorne1 50	65	70	69
GY-70	43	35	37
VS8-160	41	28	39

TABLE VIII

AVERAGE FRICTION COEFFICIENT AT 573K

Fiber	Resin Composition (%PMDA/%BTDA)		
	100	80/20	60/40
Thorne1 50	0.24	0.21	0.22
GY-70	0.29	0.18	0.14
VS8-160	0.30	0.19	0.25

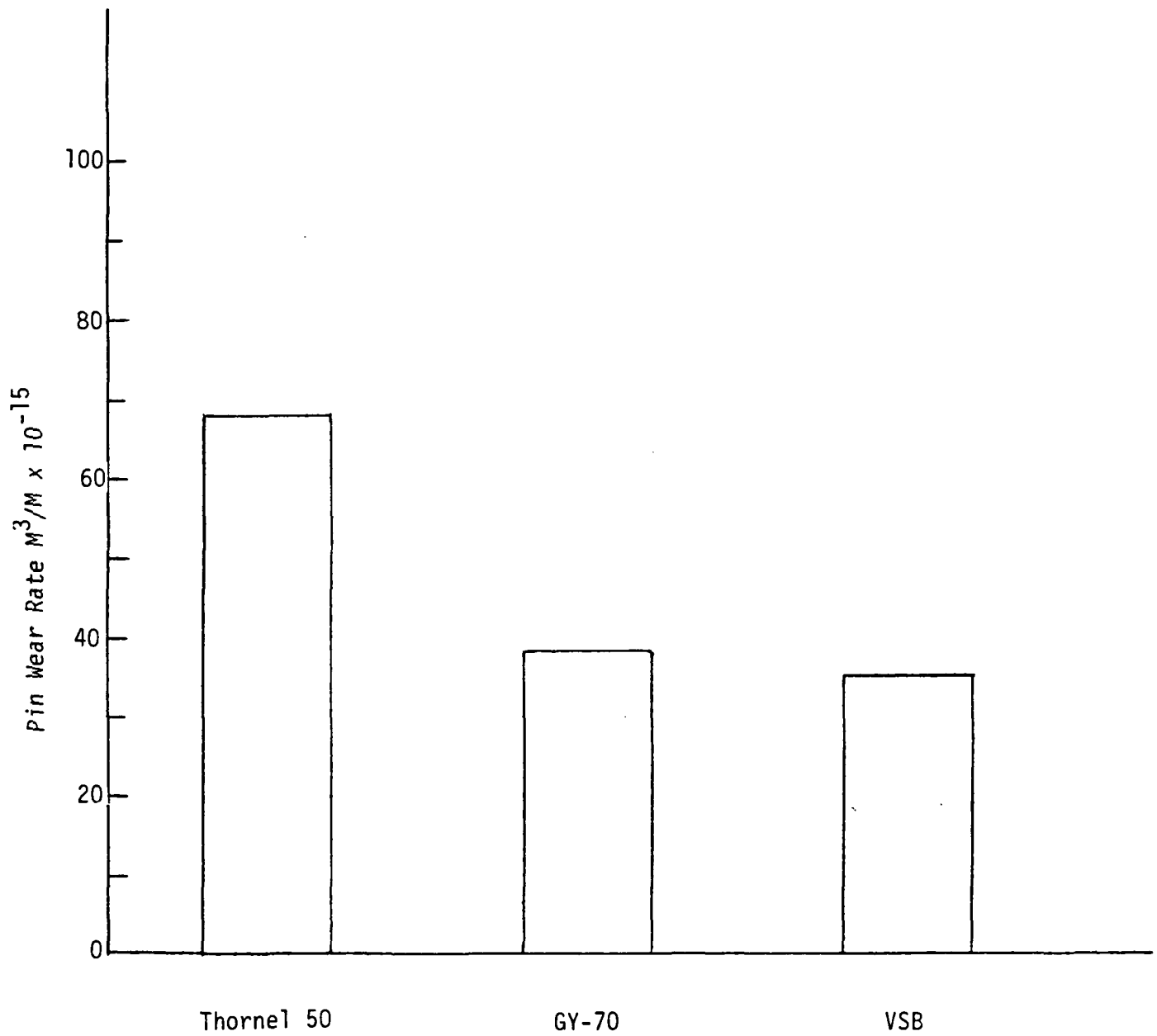


Figure 10. Arithmetic average of wear rates at 573K on all composite pins.

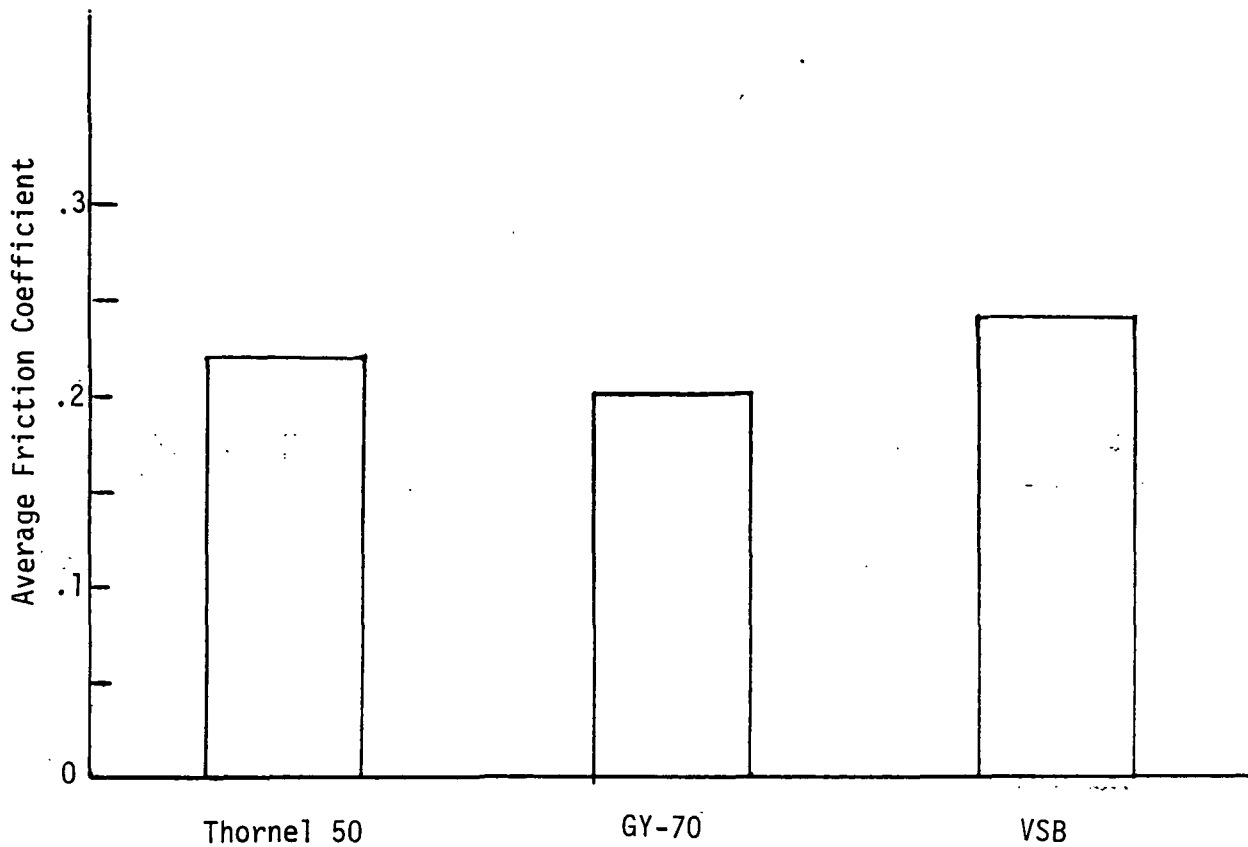


Figure 11. Arithmetic average of the friction coefficients obtained at 573K for all the composite pins.

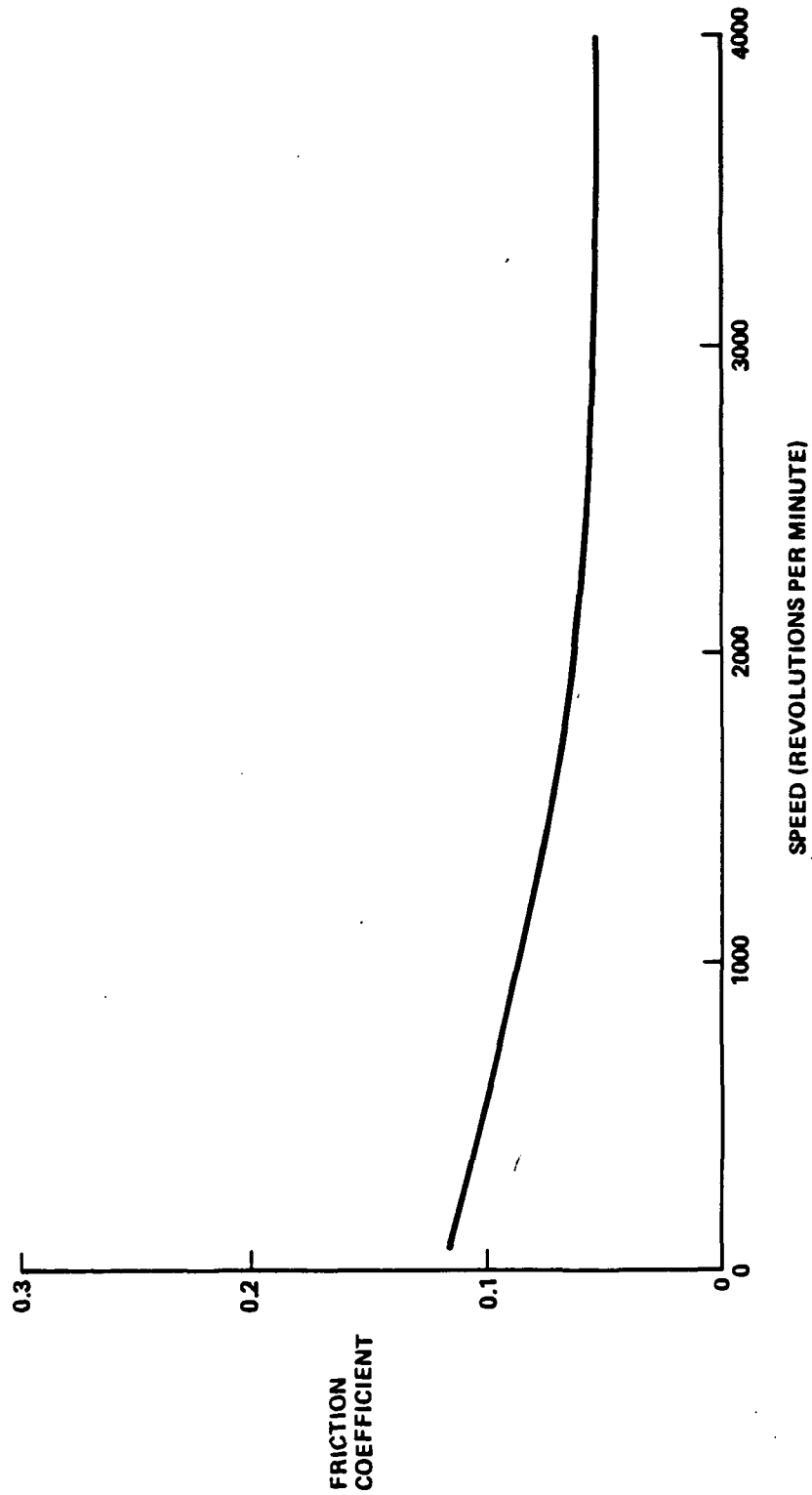


Figure 12. Friction Coefficient vs. Speed at 573K of 4-BDAF/PMDA Polyimide Reinforced with 50% by Weight GY-70 Chopped Graphite Fibers



## 6.0 CONCLUSIONS AND RECOMMENDATIONS

### 6.1 Conclusions

On the basis of the tribological tests performed on these non-optimized test specimens, it appears that the carbon fiber filled PFPIs, particularly the 80/20 compositions, show an excellent balance of wear/friction at 573K. These results are very promising in the context of an apex seal for advanced rotary engines.

The results of the tests with 80/20 and 60/40 unfilled resin compositions at 298K are intriguing for their unusual high friction/low wear properties. R.L. Fusaro has pointed out, in private communications that this combination of properties may be advantageous, in such applications as brakes and traction drives.

### 6.2 Recommendations

The badly voided nature of some of the molded samples clearly indicate that additional work on optimizing the cure and molding conditions is in order. It is possible that improved results might be obtained with more uniform specimens.

It is also recommended that additional studies be performed to determine the optimum loading of carbon fiber.

A systematic study of the relationship of carbon fiber source, mechanical properties and morphology on tribological behavior would also be of value in providing the best performance in a rotary engine apex seal.

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